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## Composition of Syntroleum S-5 and Conformance to JP-5 Specification

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The U.S. Military uses JP-8/JP-5/Jet A-1 fuel. JP-8 is identical to Jet A-1 except that it contains additives required by the military. JP-5 differs from JP-8 only in that it has a slightly higher flash point. A synthetic JP-5 fuel produced by Syntroleum Corporation, S-5, differs from petroleum-derived jet fuels in that it has a much lower normal paraffin concentration and it does not contain any detectable S, N, or O compounds, or aromatics. Despite these compositional differences, S-5 conforms to all JP-5 specification requirements for the chemical and key physical properties evaluated, except the density; S-5 density was slightly lower than the minimum density required. One other key requirement for JP-5 states that, "The feed stock from which the fuel is refined shall be crude oils derived from petroleum, tar sands, oil shale, or mixtures thereof." Fuels such as S-5, synthesized from feed stocks other than those allowed, do not conform to the JP-5 specification in this regard.

# COMPOSITION OF SYNTROLEUM S-5 AND CONFORMANCE TO JP-5 SPECIFICATION

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## Introduction

As the U.S. Military anticipates fuel requirements for far into the future, and at the same time considers global sources for those fuels, fuels produced via non-conventional means are forecasted to become increasingly available. One such type of fuel, a synthetic fuel, can today be produced from a synthesis process first developed in the 1920's known as Fischer-Tropsch. As a matter of fact, some limited production of coal-derived synthetic fuel has been a reality since the early 1970's in South Africa, and starting in the early 1990's synthetic fuel derived from natural gas has been produced in Bintulu, Indonesia. The possibility for the use of synthetic fuels by the U.S. Military opens up a whole new dimension in considering sources of supply. Various scenarios for producing synthetic fuel can be envisioned that could lead to increased energy security and enhanced mobility for the U.S. Military.

Today's military uses JP-8/JP-5/Jet A-1 fuel, designated as the Single Fuel for the Battlefield. JP-8 is identical to Jet A-1, the commercial industry standard for aviation fuel available worldwide, except that JP-8 contains three additives required by the military. JP-5 is used in naval aircraft and differs from JP-8 only in that it is produced to have a slightly higher flash point for added safety for shipboard use.

This paper describes the composition of a synthetic JP-5 fuel, S-5, produced by Syntroleum Corporation using Fischer-Tropsch synthesis. S-5 fuel was distributed among the Army, Air Force, Navy, and DOE Laboratories participating under a Memorandum of Agreement to evaluate synthetic fuels for potential use by the military. In addition, this paper will discuss the conformance of S-5 to the JP-5 specification.<sup>1</sup>

## Composition of Syntroleum S-5

The compositional data reported herein was developed by the National Energy Technology Laboratory. A significant amount of compositional data was also developed by Syntroleum Corporation, but that data is not included.

**Carbon Characterization.** Results of GC-FID analysis of S-5 for the relative amounts of normal and isoparaffins versus carbon number are given in Table 1. S-5 fuel is made up of paraffins mainly in the C-11 to C-17 range with the greatest concentration in the C-14 to C-15 range. Normal paraffins were greatest in the C-10 to C-11 range. A comparison with Jet A, JP-8 and JP-5 fuels showed that those fuels had normal paraffin concentrations that peaked in the same range or up to C-12. The main difference between the

fuels was that S-5 had a normal paraffin concentration of only 6.6% while the petroleum-derived jet fuels had normal paraffin concentrations of around 30%.

Table 1. S-5 Branched/Normal Carbon Analysis by GC-FID

	% Abundance (% Std. Dev.)			
	Normal	(Std. Dev.)	Branched	(Std. Dev.)
<C8	--	--	0.01	(0.00)
C8	0.01	(0.00)	--	--
C9	0.07	(0.00)	0.04	(0.01)
C10	1.47	(0.04)	1.02	(0.03)
C11	1.45	(0.02)	10.52	(0.17)
C12	1.14	(0.00)	11.66	(0.06)
C13	0.68	(0.00)	12.29	(0.02)
C14	0.57	(0.00)	12.81	(0.04)
C15	0.42	(0.01)	12.94	(0.05)
C16	0.53	(0.00)	12.21	(0.06)
C17	0.27	(0.00)	9.02	(0.05)
>C17	--	--	10.88	(0.13)
Total	6.61		93.39	

\* Results an average of 3 separate measurements.

**Branching Studies.** The analysis was conducted in the C-13 to C-16 range because that is the range in which slightly greater than 50% of the isoparaffins fall. The GC-MS separation proved to be fairly easy, but assigning the peaks to specific chemical structures proved difficult due to a limited spectral library and the number of isomers that could be assigned to a specific mass. Despite this uncertainty, certain conclusions could be drawn. It appeared that by far most of the isoparaffins were methyl substituted with considerably and progressively less substitution by ethyl and longer chain groups. Additional information should be possible with NMR analysis.

**Heteroatom Characterization.** GC-AED was used to analyze the sulfur, nitrogen and oxygen components of S-5. Those results were compared with analyses for the same elements in typical JP-5, JP-8 and Jet-A fuels. As expected, S-5 did not contain detectable S (1 ppm limit), N (10 ppm limit) or O (25 ppm limit) compounds. Conventional jet fuels that were analyzed were also found to contain no detectable nitrogen or oxygen compounds. It is known that those compounds are found in jet fuels, but the limits of detection when injecting a neat sample are much too high to be able to measure those compounds with GC-AED. Because of their much higher concentration in jet fuel, sulfur compounds were easily detected in the conventional jet fuels. The jet fuels typically contained the same types of sulfur compounds, but in different relative proportions depending on the fuel.<sup>2</sup>

## Conformance of S-5 to JP-5 Specification

Table 2 provides a summary of the chemical and physical requirements for JP-5, in addition to associated data for the single lot of S-5 evaluated. A number of different ASTM test methods were performed by the Army Lab and Syntroleum to determine the properties for S-5.<sup>3</sup>

**Chemical Properties.** The acidity of S-5 was determined to be 0.0014 mg KOH/g by Total Acid Number per ASTM D664, well within the JP-5 specification of 0.015 mg KOH/g max. Note, however, that the specification calls out ASTM D3242 for this determination rather than the ASTM D664 method.

The aromatic content of S-5 determined by the Army was <1 vol % per GC-FID performed on fractions separated using HPLC, while Syntroleum found the aromatic content to be 0.4 vol % when determined by ASTM D1319. The JP-5 specification allows for up to 25 vol % aromatics as determined by the latter method.

The sulfur content of S-5 was found to be <1 ppm per ASTM D5185. Syntroleum also found sulfur content to be negligible, <1 ppm, when measured by ASTM D5453. The amount of sulfur allowed in JP-5 is up to 4000 ppm.

The JP-5 specification for hydrogen content is a minimum of 13.4 mass % as determined only by ASTM D3701. Syntroleum reported a hydrogen content of 15.9 mass % for S-5 using ASTM D5291.

**Key Physical Properties.** The Army and Syntroleum results show very similar distillation curves generated per ASTM D86 for S-5. These results indicate that S-5 easily meets the JP-5 specification which calls a maximum of 206°C at 10% recovered and 300°C at the end point as measured by the same method. The temperatures at 10% recovered are 194°C and 195°C for the Army and Syntroleum respectively; and similarly the temperatures at the end point are 267°C and 271°C respectively. The D86 distillation residue and loss limits in the JP-5 specification are 1.5 vol % maximums and S-5 residue and loss are under both these limits as reported by the Army and Syntroleum.

The JP-5 flash point specification is 60°C minimum and both labs, using different but allowed methods, found the S-5 flash point to be slightly above this minimum.

The density range allowed in the JP-5 specification is 0.788 – 0.845 kg/L at 15°C. Syntroleum, using ASTM D4052 (Referee Method), determined the density of S-5 at 15°C to be 0.764 kg/L. The Army, using ASTM D1298, also determined the density of S-5 at 15°C to be 0.764 kg/L. The S-5 does not meet the density requirement for JP-5.

The freezing point called out in the specification is for a maximum of -46°C per ASTM D5972. As measured with this same test method, the Army determined a freezing point of -50°C while Syntroleum reported a value of -51°C.

Both the Army and Syntroleum measured the kinematic viscosity at -20°C per ASTM D445. A maximum of 8.5 mm<sup>2</sup>/s is called out in the JP-5 specification and both the Army and Syntroleum reported values of 6.0 mm<sup>2</sup>/s and 6.1 mm<sup>2</sup>/s, respectively, which easily meet this limit.

The net heat of combustion of S-5 determined by Syntroleum was 44.1 MJ/kg using ASTM D4529 which meets the JP-5 specification of 42.6 MJ/kg minimum.

Syntroleum measured the smoke point of S-5 to be >43 mm per ASTM D1322, greatly surpassing the JP-5 specification calling for a minimum of 19.0 mm.

**Other Physical Properties.** Several other physical requirements are dictated in the JP-5 specification as

summarized in Table 2. These include corrosivity limits, thermal stability requirements, contaminant limits, and microseparator rating limits for fuel containing various combinations of the mandatory additives. S-5 has not yet been evaluated for these properties; a more complete assessment of the conformance to the JP-5 specification needs to include them.

## Conclusions

Several analytical techniques and ASTM test methods were used to determine the composition, chemical and physical properties of a synthetic JP-5 fuel, S-5, produced by Syntroleum Corporation. S-5 differs from petroleum-derived jet fuels in that it has a much lower normal paraffin concentration and it does not contain any detectable S, N, or O compounds, or aromatics. Despite these compositional differences, S-5 conforms to all JP-5 specification requirements for the chemical and key physical properties discussed previously, except the density; S-5 density was slightly lower than the required minimum density.

One other key requirement in the JP-5 specification not previously mentioned is that, "The feed stock from which the fuel is refined shall be crude oils derived from petroleum, tar sands, oil shale, or mixtures thereof." Fuels such as S-5, synthesized from feed stocks other than those allowed, do not conform to the JP-5 specification in this regard. However, specifications for Jet A-1, U.K. Defence Standard 91-91 used by foreign military and the commercial airline industry worldwide, and ASTM D1655 used by the commercial airline industry in the U.S., have made some allowance for fuels containing synthetic components derived from non-petroleum sources, but only with strict adherence to very specific requirements, including assessments of fit for purpose.<sup>4</sup>

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## References

- (1) Military Specification, MIL-DTL-5642T, "Turbine Fuel, Aviation, Grades JP-4, JP-5, and JP-5/JP-8 ST", September 1998.
- (2) Link, D.D.; Baltrus, J.P.; Rothenberger, K.S.; Zandhuis, P.; D. K. Minus, D.K., Striebich, R. C. "Class- and Structure-Specific Separation, Analysis and Identification Techniques for the Characterization of the Sulfur Components of JP-8 Aviation Fuel" *Energy & Fuels* **2003**, 17, 1292-1302.
- (3) ASTM International, Annual Book of ASTM Standards, Section 5, Petroleum Products, Lubricants, and Fossil Fuels, West Conshohocken, PA, **2003**.
- (4) Ministry of Defence, Defence Standard 91-91, "Turbine Fuel, Aviation Kerosine Type, Jet A-1", Defence Procurement Agency, Directorate of Standardization, Kentigern House, Glasgow, U.K., June 2002.

**Table 2. S-5 Test Results vs. JP-5 Specification**

<i>Property</i>	<i>ASTM Test Method<sup>1</sup> Army (Syntroleum)</i>	<i>Limits</i>	<i>S-5 Data Actual Army (Syntroleum)</i>	<i>JP-5 Spec (MIL-DTL-5624T)</i>
<b>APPEARANCE</b>				
Saybolt color	<b>D156 (D156)</b>		+30 (+30)	report
<b>COMPOSITION</b>				
Total acid number, mg KOH/g	D664 <sup>2</sup>	max	0.0014	0.015
Aromatics, volume %	I.M. <sup>3</sup> (D1319)	max	<1 (0.4)	25.0
Sulfur, mercaptan, mass %	D3227	max	--	0.002
or Doctor test	D4952		--	negative
Sulfur, total, mass %	D5185 (D5453)	max	<0.0001 (<0.0001)	0.40
<b>VOLATILITY</b>				
Distillation temp. D 86, °C:	<b>D86<sup>4</sup> (D86)</b>			
Initial boiling point			183 (185)	report
10% recovered		max	194 (195)	206
20% recovered			201 (199)	report
50% recovered			219 (218)	report
90% recovered			254 (253)	report
Final boiling point (end point)		max	267 (271)	300
Distillation residue, volume %		max	1.3 (0.6)	1.5
Distillation loss, volume %		max	0 (1.2)	1.5
Flash point, °C:	D56 <sup>5</sup> ( <b>D93</b> )	min	62 (64)	60
Density at 15°C, kg/L	D1298 ( <b>D4052</b> )	min-max	0.764 (0.764)	0.788-0.845
<b>FLUIDITY</b>				
Freezing point, °C:	D5972 (D5972)	max	-50 (-51)	-46
Viscosity - 20°C, mm <sup>2</sup> /s:	D445 (D445)	max	6.0 (6.1)	8.5
<b>COMBUSTION</b>				
Net heat of combustion, MJ/kg	(D4529)	min	(44.1)	42.6
Calculated Cetane Index	D976 <sup>6</sup>		69.5/67.3 <sup>7</sup> (68.7)	report
Hydrogen content, mass %	(D5291 <sup>8</sup> )	min	(15.9)	13.4
Smoke point, mm	(D1322)	min	(>43)	19.0
<b>CORROSION</b>				
Copper strip, 2 h at 100°C, rating	D130	max	--	1
<b>THERMAL STABILITY</b>				
Filter pressure drop, mm Hg	D3241 <sup>9</sup>	max	--	25
Tube deposit code	D3241 <sup>9</sup>	max	--	3 <sup>10</sup>
<b>CONTAMINANTS</b>				
Existent gum, mg/100 mL	D381 <sup>11</sup>	max	--	7.0
Particulate matter, mg/L	D2276 or <b>D5452</b> <sup>12</sup>	max	--	1.0
Filtration time, minutes	D2276 or <b>D5452</b> <sup>12</sup>	max	--	15
Water reaction: Interface rating	D1094 <sup>12</sup>	max	--	1b
<b>OTHER</b>				
Micro-separometer ratings:	D3948			
With AO <sup>13</sup> , MDA <sup>13</sup>		min	--	90
With AO <sup>13</sup> , MDA <sup>13</sup> & FSII <sup>13</sup>		min	--	85
With AO <sup>13</sup> , MDA <sup>13</sup> & CI/LI		min	--	80
With AO <sup>13</sup> , MDA <sup>13</sup> , CI/LI & FSII		min	--	70
Fuel system icing inhibitor, vol %	D5006 <sup>14</sup>	min-max	--	0.15-0.20
<b>Notes:</b> <sup>1</sup> Referee method in bold. <sup>2</sup> MIL-DTL-5624T calls for ASTM D3242. <sup>3</sup> Internal Method using HPLC separation technique followed by GC-FID. <sup>4</sup> A condenser temperature of 0°C to 4°C shall be used for the distillation. <sup>5</sup> ASTM D56 may give results up to 1°C below the ASTM D 93 results. <sup>6</sup> If D86 values are used to calculate Cetane Index, correct to standard barometric pressure. <sup>7</sup> Results shown for Equation 1/Equation 2 calculations. <sup>8</sup> MIL-DTL-5624T allows use of ASTM D3701 only for determination of hydrogen content of JP-5. <sup>9</sup> Conditions: heater tube max. point T = 260°C; fuel P = 3.45 MPa, flow rate = 3.0 mL/min.; time = 150 min. <sup>10</sup> Peacock or abnormal color deposits result in a failure. <sup>11</sup> If air used instead of steam, must report. In case of failure with air, retest using steam. <sup>12</sup> Sample size min. = 3.79 liters; determine filtration time per Appendix A, MIL-DTL-5624T. <sup>13</sup> Samples submitted for conformance testing shall contain same additives present in refinery batch; the refiner shall report rating on laboratory hand blend of fuel with all additives required by specification. <sup>14</sup> The DIEGME scale of the refractometer shall be used when completing this test.				